SPECIFICATION

PRODUCING METHOD AND PRODUCING APPARATUS OF NUCLEAR SPIN POLARIZED XENON GAS

TECHNICAL FIELD

This invention relates to producing method and producing apparatus of nuclear spin polarized xenon gas, and more specifically, relates to producing method and producing apparatus of nuclear spin polarized xenon gas capable of producing a polarized nuclear spin useful for NMR•MRI apparatus with high concentration and continuously.

BACKGROUND

It has been recently reported that when xenon gas with a nuclear spin polarized (nuclear spin polarized xenon gas) is applied to NMR•MRI method, detection sensitivity is enhanced rapidly.

Being polarized termed herein is that distribution of the spin number occupying energy order of nuclear spins of an atomic nucleus corresponding to the orientation state with respect to the metal static magnetic field is extremely one-sided.

Rare gas having the polarized state is obtained in the procedure such that circularly polarized excitation light is irradiated on gas having rare gas including a single atomic molecule having a nuclear spin of spin quantum number 1/2 such as xenon-129 (^{129}Xe), helium-3 (^{9}He) or the like mixed with alkali metal vapor such as rubidium, cesium or the like whereby electron in the base state order of rubidium or the like is excited by light absorption in the base state order and returned to the base state order after passage of the base state order, at which time it is transited with

high probability to one order of electron orders out of the base state orders whose degeneracy is released magnetically by the magnetic field applied from outside to prepare the state that an electronic spin polarization degree of a rubidium molecule or the like is high, and the rubidium or the like in the high polarization state collides with rare gas such as xenon, at which channel the high polarization state of rubidium or the like moves to a nuclear spin system of rare gas such as xenon. This channel is generally called optical pumping.

In the conventional polarized rare gas producing apparatus, a mixed gas of rare gas and alkali metal vapor is confined into an optical reaction vessel, to which irradiation of excited light and application of magnetic field are carried out. For example, there is a producing apparatus, for the purpose of using polarized helium-3 of high density as a neutral polarizer, in which a mixed gas of helium-3 gas and nitrogen gas and alkali metal are confined into a cylindrical glass ample (for example, see M.E. Wagshul and T.E. Chu PP, Phy, Rev. A40, 4447 (1989)).

On the other hand, there is an apparatus in which for example, 1% of xenon is mixed with buffer gas of helium of 10 atmospheric pressure or so, introduced into a cylindrical glass vessel, irradiated, polarized, and guided into Dewar cooled by liquid nitrogen from a gas outlet of the vessel, and polarized xenon is formed into a solid, which is separated whereas the remaining helium gas is discharged from a vent line (For example, see B. Driehuys, G.D. Cates, E. Miron, K. Sauer, D.K. Walter and W. Happer, Appl. Phys, Lett. 69, 1668(1996).

In any of those apparatuses as noted above, operation for enhancing the polarization rate is carried out by receiving a laser beam in the state that rare gas or the like is stayed in an optical reaction vessel. When the

polarization rate was enhanced, the temperature is cooled to a room temperature, and being used as a neutral polarizer as it is, or polarized xenon 129 once solid-separated within Dewar is heated again into gas, and transferred to a separate vessel for use in measurement of NMR or the like.

However, in the above-mentioned conventional method, for facilitating polarization, xenon is diluted, for example, to helium 2% xenon concentration or so and polarized, gas containing produced xenon is frozen with liquid nitrogen, which is heated to remove only the xenon to produce high concentration xenon gas, thus posing a problem that work efficiency is extremely poor. In addition, in the conventional apparatus in which gas or the like is stayed and polarized, since polarized gas cannot be generated continuously, polarized gas is taken out into a separate vessel every time and carried to NMR apparatus, thus taking time, and posing a problem that the polarization rate reduces during such a period of time as described.

This invention has been accomplished in view of the foregoing, and has its object to provide producing method and producing apparatus capable of obtaining polarized xenon gas of high concentration without being frozen, and capable of generating polarized xenon gas continuously.

It is a further object of this invention to provide producing method and producing apparatus of a glass cell in which metal rubidium and xenon gas used for the above-described producing method and producing apparatus are solidified and sealed under the absence of oxygen.

DISCLOSURE OF THE INVENTION

The producing method according to Claim 1 out of the inventions suited for the aforementioned objects is characterized in that a glass cell having solid rubidium and solid xenon filled in the pressure reducing state

of being absent in oxygen is heated to be gas xenon and gas-liquid mixed rubidium, to which a magnetic field is applied to irradiate a laser beam. It is noted that being absent in oxygen termed herein is meant not to oxidize solid rubidium, and the presence of a fine amount of oxygen to a degree that even if solid rubidium is oxidized, reaction is not affected, is allowed.

When the thus produced nuclear spin polarized xenon gas is taken out, pressure naturally lowers and air flows backward into the glass cell. Therefore, xenon polarized gas is taken out while introducing xenon gas so as to maintain fixed pressure. Further, by doing so, polarized xenon gas can be produced continuously.

Preferably, it is constituted so that in replacing a xenon gas supply device, the xenon gas supply device side is made to be a primary side through a first air operate valve, and the xenon gas introducing side of the glass cell is made to be a secondary side, and vacuuming of the primary side piping and pressurization-leaving by nitrogen gas are repeated automatically more than three times.

Preferably, in replacing the glass cell, vacuuming of piping from the primary side piping, the secondary side pipe and the primary side pipe to a valve on the polarized xenon gas take-out side communicated through a second air operate valve and pressurization-leaving by nitrogen gas are repeated automatically more than three times.

A producing method of a glass cell according to the present invention is characterized in that a chamber housing therein rubidium filled into a glass vessel and said glass cell are connected so that they are communicated by piping, said piping is exhausted by a vacuum generator, after which a glass vessel filled with rubidium is broken to heat metal rubidium, piping and glass cell, rubidium of gas is made present within the

piping and glass cell, then said glass cell is cooled, metal rubidium is precipitated as a solid into the cooled portion, xenon gas is introduced into the glass cell and closed, and the glass cell is cooled to solidify xenon within the glass cell.

A producing apparatus of the present invention is characterized by comprising means for heating a glass cell having solid rubidium and solid xenon filled in the pressure reducing state of being absent in oxygen to be gas xenon and gas-liquid mixed rubidium, and means for applying a magnetic field to the glass cell to irradiate a laser beam.

Further, preferably, there comprises means introducing xenon gas while taking out the produced nuclear spin polarized xenon gas, and pressure regulating means for controlling said operation so that pressure may not drop.

Preferably, a xenon gas supply device side is made to be a primary side piping through a first air operate valve, piping extended up to a valve for introducing xenon gas into a glass cell is made to be a secondary side piping, branched pipings connected to said primary side piping through a second air operate valve, one of said branched pipings reaching a vacuum generator and the other reaching a valve on the xenon gas taking-out side of said glass cell, and pressure regulating means for regulating pressure introduced into the glass cell is provided on the primary side piping.

A producing apparatus of a glass cell according to the present invention comprises piping connected so that a chamber housing rubidium filled into a glass vessel and the glass cell are connected, means for vacuuming the piping, means for breaking glass having rubidium filled in, means for heating metal rubidium, piping and a glass cell, and means for cooling the glass cell and precipitating metal rubidium on the cooled

portion.

In summary, the gist of the present invention lies in that xenon is filled into the glass cell having metal rubidium adhered thereto heated to irradiate a laser beam thereby obtaining polarized xenon gas of high concentration without being frozen.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic sectional view showing a producing apparatus of a glass cell having rubidium and xenon filled therein according to the present invention.

FIG. 2 is a sectional view showing the state that rubidium and xenon are filled in, and thereafter a glass cell is closed and sealed.

FIG. 3 is a structural view showing one embodiment of the producing apparatus according to the present invention.

BEST MODE FOR CARRYING OUT THE INVENTION

In the following, the embodying form of the present invention will be described with reference to the drawings.

FIG. 1 shows a producing method (apparatus) of a glass cell having rubidium and xenon filled in, in which a vacuum pump 5 and a xenon gas supply line 6 are connected to piping 4 connecting a metal rubidium 1 enveloped with glass housing chamber 2 and a glass cell 3. When rubidium comes in contact with air, it is oxidized and changed into oxide of rubidium, and therefore in case of purchasing it from makers, it is filled in glass as described above. It is noted that in this state, a valve Vxe, a valve V1 and a valve V2 are closed.

If a valve Vp is opened, in the state that the valve Vxe is closed, to

exhaust by the vacuum pump 5, air in the piping 4 and the glass cell 3 is exhausted. When, in this state, glass in which rubidium is filled is broken, metal rubidium will be present in vacuum, thus not being oxidized. A magnet enveloped with glass is also filled in the metal rubidium 1 housing chamber 2, which is moved by the magnet from outside to be placed in contact with metal rubidium 1 to break glass.

Next, the metal rubidium 1, glass cell 3 and piping 4 are wholly heated. It is suggested that a heating temperature is not less than a melting point (about 40°C) of rubidium, and is a temperature that rubidium assumes a gas-liquid mixed state in which liquid and gas of a vapor pressure portion at that temperature are present with high concentration. More specifically, preferably, 130 ~180°C, particularly preferably, near 150°C.

Then, when only a part of glass cell is cooled, rubidium in the gas state is solidified on only the cooled part and separated as shown in FIG. 3.

Then, when the vacuum pump 5 side valve Vp is closed and Vxe is opened, gaseous xenon is introduced into the glass cell 3. Next, when Vxe is closed, and the whole glass cell 3 is cooled with liquid nitrogen, xenon is turned to solid. Originally, since xenon is introduced into solid rubidium confined in vacuum, if xenon is solidified, the glass cell assumes a pressure reducing state (vapor pressure at a liquid nitrogen temperature).

In this state, if a portion 8 depicted in FIG, 2 is heated and molten by operation of a burner, the glass cell 3 having solid rubidium and solid xenon filled in is obtained.

Using the glass cell, and there is constituted as shown in FIG. 3, and when the temperature of the glass cell 3 is elevated, preferably, to 50 ~180°C, particularly preferably, near 120°C, the glass cell will internally be xenon gas and gas-liquid mixed rubidium. In this state, if a magnetic field

is applied to irradiate laser, xenon gas will be nuclear spin polarized xenon gas in scores of minutes.

Then, as shown in FIG. 3, the valves V2 and V3 are opened, and polarized xenon gas is collected by a polarized xenon gas collecting cylinder 9, and at the same time, the valve V1 is opened to introduce xenon gas while regulating pressure by an auto pressure regulator (APC) 10 so that pressure may not drop. When polarized xenon gas in the glass cell 3 is taken out, pressure lowers and the air back flows, because of which the pressure-regulate xenon gas is introduced as described above. It is noted that in this state, air operate valves AV6, AV1 and AV3, and valves V1. V2 and V3 in FIG. 3 are opened.

Then, the valves V1 and V2 are closed, the whole glass cell 3 is cooled with liquid nitrogen, and after xenon is solidified, the glass cell 3 is heated to produce polarized xenon gas by the same operation as mentioned above. In this manner, it can be produced repeatedly continuously till rubidium as a catalyst is gone.

As shown in FIG. 3, xenon gas from a xenon cylinder11 is introduced from the valve V1 of the glass cell 3 passing though an air operate valve (AV1), an auto pressure regulator (APC) 10 and a first air operate valve (AV3). In this embodiment, since pressure for taking out polarized xenon gas is 1.5 atmospheric pressure or so, pressure of xenon gas is regulated to the same 1.5 atmospheric pressure or so by APC10. In FIG. 3, valves V1 ~ V4 are constituted by glass valves because polarized gas comes in contact, and because if glass is not employed, polarized xenon gas returns to xenon gas. Accordingly, other portions within piping with which polarized xenon gas contacts are also glass (pyrex).

Nitrogen gas and xenon gas are dropped down to pressure of 1.5

atmospheric pressure or so by pressure reducing valves (REG 2 and REG 1), respectively.

In the above-described reaction, it is necessary that air may not enter at all the glass cell. Because even if a small amount of air is mixed, the rubidium catalyst is oxidized, resulting in not exhibiting the catalyst function.

Air is mixed in at the time of replacing a cylinder and at the time of replacing a glass cell, in which case, air is prevented from mixing into the glass cell in the following manner.

In case of replacing a xenon cylinder, air is mixed into piping between an original valve 13 of the cylinder, an air operate valve (AV1) and an air operate valve (AV6). For removing the air, a vacuum pump (P) 15 is turned on, an air operate valve (AV1) and air operate valve (AV2) and a second operate valve (AV4) are opened to vacuum piping on the primary side, and the state is left as it is for fixed time while detecting a pressure reducing degree by a pressure transmitter ((PT1). Through the first air operate valve (AV3), the xenon gas supply device side is made to be a primary side, and the xenon gas introducing side of the glass cell is made to be a secondary side.

Then, the second air operate valve (AV4) is closed to pressurize he interior of piping on the primary side with nitrogen gas. The pressure on the primary side is left for fixed time preset while detecting it by the pressure transmitter (PT1). Then, the step is again repeated for vacuuming again the interior of piping on the primary side and pressurizing the interior of piping on the primary side with nitrogen gas to leave it. Preferably, it is possible to prevent oxygen from mixing into the glass cell by repeating the step more time 10 times.

When the glass cell is replaced, air is mixed into the piping between piping to the manual valve V1 at a glass cell inlet on the secondary side in communication with piping on the primary side through the first air operate valve (AV3), the manual valve V2 for taking out polarized gas, the valve V3 or controlling the flow into the collecting potion, and the valve V4 for controlling the communication with the vacuum pipe.

The valve V4 and the second air operate valve (AV4) (controlling communication between piping on the primary side and he vacuum pump) are opened to vacuum the interior of piping of the air mixing portion. Then, the first air operate valve from the both cylinder11,12 (AV3) connecting pipings on the primary side and secondary side, and the valve to the first air operate valve (AV3) are opened to pressurize piping on the primary side and piping on the secondary side with nitrogen gas, and all the valves are closed for pressurization-leaving. Then, the step is repeated for opening the first air operate valve and the second air operate valve, vacuuming similar to that mentioned above, and pressurization-leaving. Preferably, it is possible to prevent oxygen from mixing into the glass cell by repeating the step more than 10 times. Since the pressure transmitter (PT2) is disposed on piping on the secondary side, vacuuming and pressurization-leaving are performed for fixed time pre-set while sensing pressure. The air operate valve (AV5) in FIG. 3 is a valve for releasing gas when the interior of the primary piping is in the pressurizing state, which is however not used in the above-described operation.

In FIG. 3, while nitrogen and xenon gas are supplied from the cylinder, well known other gas supply devices will suffice.

According to the present invention, reaction is done with high concentration such as xenon gas 80 ~ 100% (remainder, nitrogen gas) to

thereby obtain polarized xenon gas, thus obtaining polarized xenon gas of high concentration without carrying out treatment such as solidifying after being polarized.

Further, vacuuming the interior of piping and pressurization-leaving can be carried out many times to prevent air from flowing into the glass cell.

As described above, according to the present invention, since xenon gas of high concentration can be used to produce polarized xenon gas of high concentration, trouble of concentrating by freezing after production as in prior art can be eliminated.

Further, xenon gas as raw material is regulated in pressure and introduced while taking out polarized xenon gas, whereby back-flow of air is prevented, and polarized xenon gas can be produced continuously.

Furthermore, vacuuming the interior of piping and pressurization-leaving are carried out repeatedly to thereby sufficiently enable purging the interior of piping, and so, it is possible to prevent mixing of air in he reaction glass cell, and to extend the life of the rubidium catalyst.

Accordingly, it is expected to utilize the invention as producing method and producing apparatus of producing polarized nuclear spin polarized xenon gas useful for NMR•MRI apparatus with high concentration and continuously.